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STATUS REPORT

MEASUREMENT OF REFLECTIVITIES OF ROCKS, MINERALS
AND GLASSES IN THE VACUUM ULTRAVIOLET

NSG-9056

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Enclosed are copies of two papers by the principal investigator and his colleagues entitled, "Reflectance Measurements of Lunar Materials in the Vacuum Ultraviolet" and "Vacuum Ultraviolet Spectra of Carbonaceous Chondrites." These papers constitute the status report. Our progress to date is also described in the progress report which is part of our proposal of July 31, 1979, to NASA for renewal of this grant.

Reflectance measurements of lunar materials in the vacuum ultraviolet

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Abstract—The spectral reflectances of materials have been measured in the vacuum ultraviolet region of the spectrum for possible planetary compositional remote-sensing applications. These materials, which include lunar and terrestrial silicate rocks and minerals, have one or more characteristic absorption peaks between about 8 and 13 eV. The peaks are visible in the spectra of powders and rough surfaces as well as polished surfaces. In lunar samples the most prominent peaks are at 10.25 eV, due to augite, and at 9.0 eV, due to anorthite. Spectra of lunar soils have minima between 6 eV (0.20 μm) and 8 eV (0.16 μm) depending on composition. Soils that have high reflectances in the visible have low reflectances in the VUV. The pyroxene content of the soils results in reflectance maxima at about 10 eV (0.12 μm). Peaks due to olivine and augite can be identified in the spectra of meteorites.

INTRODUCTION

Although the vacuum ultraviolet has been used for many years to study fluorescent radiation from planetary atmospheric gases, this portion of the electromagnetic spectrum has been virtually ignored for remote sensing of solid surfaces. Only a few measurements of the moon (Lucke, *et al.*, 1974, 1976) and Mercury (Wu and Broadfoot, 1977) have been carried out over a limited range of wavelengths in the far-UV. However, in this spectral region the energy of a photon is of the same order as the valence-conduction band gap in most solids of geological interest, so that reflectance spectra may contain significant compositional information.

This paper is the first report of laboratory measurements designed to study systematically the reflectance spectra of terrestrial, lunar and meteoritic samples in the VUV. Previous workers have measured reflectances of polished surfaces of selected minerals (e.g., Philipp, 1971; Harlow and Shankland, 1974; Nitsan and Shankland, 1976). However, since most airless solar system objects appear to be covered with a regolith, we have studied powdered materials as well as polished surfaces.

EXPERIMENTAL

A schematic diagram of the apparatus used to make the reflectance measurements is shown in Fig. 1. The light source is a windowless hydrogen discharge lamp which is separated from the spectrometer by two stages of differential pumping. The monochromator is a Seya-Namioka type, commercial Jarrell-Ash instrument with a holographic grating and trapped diffusion pump. A concave SiC mirror focuses the exit slit of the monochromator onto either the detector or the sample. The detector consists of a

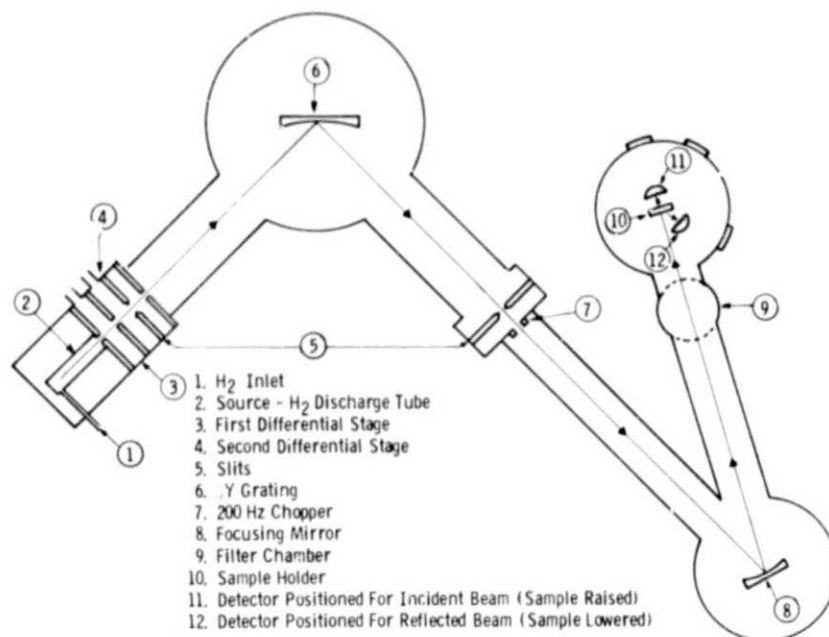


Fig. 1. Schematic diagram of reflectance apparatus.

fluorescent sodium salicylate coating on the end of a light pipe whose other end is optically coupled to a photomultiplier tube. Photon-counting electronics are used. The light pipe and sample can be rotated so that the detector looks either directly at the incident beam or at light scattered from the sample in a specular geometry with a phase angle of about 15°.

The sample surface is oriented vertically, and can be either a polished surface, a rough chip or a powder pressed into a cup. If the surface is polished, the reflected beam remains collimated and is completely sampled by the detector, so that the ratio of reflected-to-incident light is an absolute measurement of reflectivity. However, for rough surfaces and powders the detector samples only a portion of the reflected light, which is scattered into all directions, so that the measurement is relative. Absolute calibration of the reflectance of a rough surface is difficult because of the uncertainties in the photometric function of the sample and changes in sensitivity near the edges of the sodium salicylate coating. Because of these difficulties, it is common practice at longer wavelengths to normalize all measurements of a rough surface to the reflectance of MgO or BaSO₄ powder, which approximates a perfect diffusing surface. However, no material is known that could be used as a Lambert surface in the VUV.

Scattering from walls and other parts of the vacuum system was found to be negligible. Based on counting statistics, the reflectances have a precision better than 2%, except for rough surfaces at photon energies beyond about 12.5 eV, where the intensity of the source is low. The range of our measurements is from about 5 to 14 eV (about 0.25 to 0.09 μm), and the spectra are sampled at 0.25 eV intervals.

The UV radiation may cause certain minerals to emit fluorescent radiation, to which our detector would be sensitive. Because fluorescent radiation is emitted into all directions, the efficiency for its detection is reduced by $\sim \Omega/4\pi$, where $\Omega \approx .04$ is the solid angle of the detector seen from the surface. However, the light specularly reflected from a polished surface remains collimated. Thus fluorescence can be neglected for polished surfaces, but could introduce spurious signals when looking at roughened

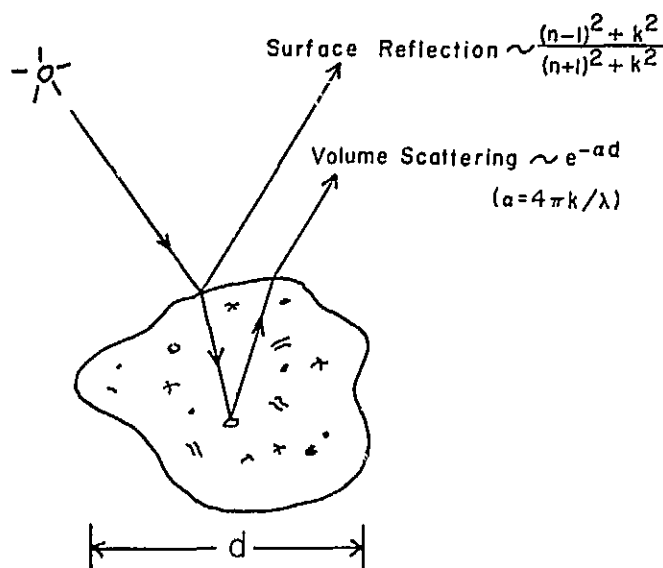


Fig. 2. Schematic diagram of processes contributing to light scattering from a powder.

or powdered surfaces, which also scatter light into all directions. For this reason, whenever possible, we measure materials in both polished and powdered form and only peaks which are observed in the reflectances of both types of surfaces are reported. Another type of spurious feature, which is visible in the spectra of certain highly-reflecting powders, is a small dip at 6.5 eV (0.19 μm) which is caused by a small amount of diffusion pump oil adsorbed onto the grain surfaces.

THEORETICAL

For a polished sample almost all of the light is specularly reflected from the front surface. At small angles of incidence and reflection the reflectivity r is given by the Fresnel equation

$$r = \frac{(n-1)^2 + k^2}{(n+1)^2 + k^2}, \quad (1)$$

where $n(\lambda)$ and $k(\lambda)$ are the real and imaginary parts, respectively, of the index of refraction.

Light can be scattered from a powder by two processes (Fig. 2): reflection from the grain surface and internal or volume scattering of rays which have been refracted into the interior of the grain and scattered or reflected back out. If the facets which make up the grain surfaces are large compared with the wavelength, the intensity of light scattered by the first process is proportional to r (Eq. 1). Since the facets are approximately randomly-oriented, the radiation will be scattered in all directions. If the grain surfaces have structures which are comparable to λ in

size, a wavelength-dependent diffuse background to the specular surface component will also be present.

The intensity of light scattered by the second process depends on the transmittance of the grain

$$t = \exp(-\alpha d), \quad (2)$$

where d is the grain size and α is the absorption coefficient

$$\alpha = 4\pi k/\lambda. \quad (3)$$

If $k \ll 1$, as is the case for non-opaque minerals at wavelengths longer than about $0.2 \mu\text{m}$, volume-scattering dominates and a reflectance spectrum is qualitatively similar to a transmittance spectrum: an absorption band causes a depression in the reflectance. If $k \approx 0.1$, the absorption length α^{-1} is shorter than a wavelength and most of the refracted component is absorbed. In this case the surface scattering dominates and, according to Eq. (1), an absorption band tends to cause a maximum in the reflectance spectrum.

In principle, r can be measured as a function of λ and the results inverted to give $n(\lambda)$ and $k(\lambda)$ separately, using the Kramers-Kronig relations (c.f., Wooten, 1972). Initially we had intended to carry out such analyses for our samples. However, an accurate absolute measurement of $r(\lambda)$ requires an extremely well-polished surface to minimize the wavelength-dependent diffuse background. We found that most of our natural samples would not take the required degree of polish because of the presence of cracks, bubbles, grain boundaries and other imperfections. Hence we abandoned the goal of Kramers-Kronig analyses, which are not essential for remote-sensing applications.

An excellent brief review of absorption processes in solids can be found in Nitsan and Shankland (1976). At long visible and near-IR wavelengths absorption in silicates is mainly by weak d-d electronic transitions (Burns, 1970). Color centers are less important in covalent materials like silicates, but can be a major cause of absorption in ionic salts. At shorter visible and near-UV wavelengths absorption is likely to be by stronger charge-transfer processes, such as $\text{Fe}^{+2}\text{-Ti}^{+4}$ or $\text{Fe}^{+2}\text{-O}^{+2}$ (Wells and Hapke, 1977). In the VUV absorption is primarily by very strong exciton (electron-hole pair) formation or valence-conduction band transitions. Since the VUV bands are due to fundamental lattice transitions they are relatively insensitive to impurities. In opaque minerals absorption can be either by charge-transfer transitions or conduction-band electrons.

MEASUREMENTS

The reflectance spectra of selected materials are shown in Figs. 3–10. Typical of polished surfaces are the spectra of MgF_2 and adularia (K-feldspar) given in Figs. 3 and 4, respectively. The reflectance of a well-polished monomineralic sample is usually low at long wavelengths because the absorption is relatively weak there, but rises to two peaks in the 8–13 eV region. The lower energy peak is usually sharper and stronger than the higher energy one. The origins of these reflectance

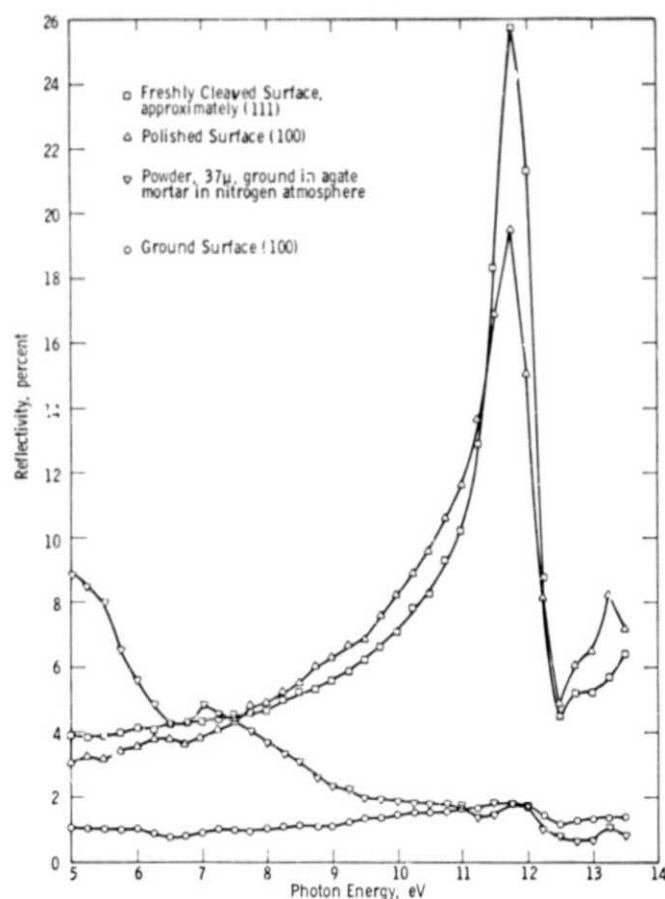


Fig. 3. Reflectance vs. photon energy for synthetic MgF_2 for several types of surface preparation.

peaks are not completely understood, but it is believed that the lower-energy band is associated with exciton formation, while the broader, higher-energy band is associated with valence-conduction band transitions (Nitsan and Shankland, 1976). Sometimes less pronounced features are visible on the low-energy flank of the first peak; these may be charge-transfer bands.

Figure 3 shows the changes which take place in the spectrum as a surface is roughened. Going from a cleaved to a polished surface causes the peaks to decrease in height and broaden slightly. As the surface is further roughened to a ground surface the apparent reflectance decreases because the reflected light is now spread out in all directions. However, the two peaks can still be observed in the spectrum, although the height of the peaks relative to the low energy wing is much smaller. The peaks remain even when the material is ground to a powder;

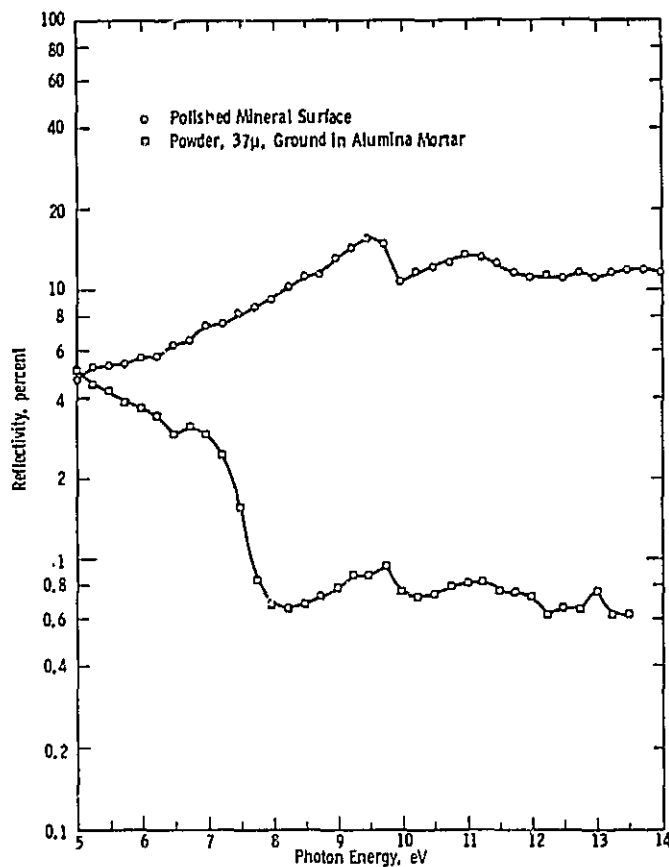


Fig. 4. Reflectance vs. photon energy for a K-feldspar (adularia), polished surface and powder. Note that the relative contrast of the absorption peaks is nearly as large in the powder as in the polished surface.

the position of the maximum may be shifted very slightly (≈ 0.25 eV) toward higher energy. The long-wavelength side of the spectrum of the powder rises considerably because of the volume-scattered light. The transition from predominantly-volume to predominantly-surface scattering is usually marked by a minimum in the reflectance spectrum. Comparison of the spectra of the polished and powdered forms of the same material shows that this transition occurs where k is about half of its peak value.

In Fig. 4 the spectrum of a polished surface is compared with a powdered surface for adularia. When plotted on a semi-log scale to preserve relative contrasts, it is seen that the ratio between the height of the first peak and the trough between the two peaks is nearly the same (about 1.3:1) for the powder as for the polished surface. This is an important result for remote compositional sensing.

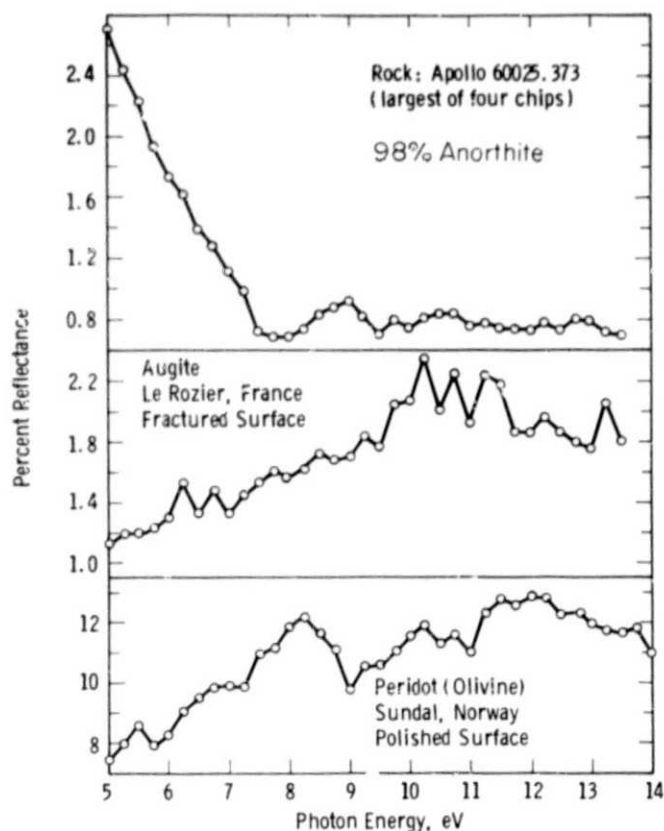


Fig. 5. Reflectance spectra vs. photon energy for several translucent minerals.

In Figs. 5 and 6 are shown the spectra of minerals commonly found in lunar materials. Figure 5 gives the spectra of a lunar anorthite, a terrestrial augite and a terrestrial olivine. Figure 6 includes terrestrial ilmenite and synthetic metallic Fe. Comparison of Figs. 4 and 5 shows that Ca-feldspar, with its main peak at 9.0 eV, can easily be distinguished from K-feldspar, which has its first band at 9.75 eV. Ilmenite has a broad maximum at 8.5 eV, while the olivine has its main band at 8.25 eV. The augite appears to have a complicated structure: it has several peaks between 10 and 12 eV, with the largest at 10.25 eV plus several minor spectral features. We have repeated these measurements on several augite samples at higher resolution and have verified this structure. The results reported here are in general agreement with those of Nitsan and Shankland (1976). These workers found that the substitution of Fe for Mg in olivine causes the first peak to shift toward lower energies.

These same bands can be seen in the spectra of lunar samples shown in Fig. 7. Rocks 14310 and 70017 both show the 9.0 eV An peak and the 10.25 eV Aug peak.

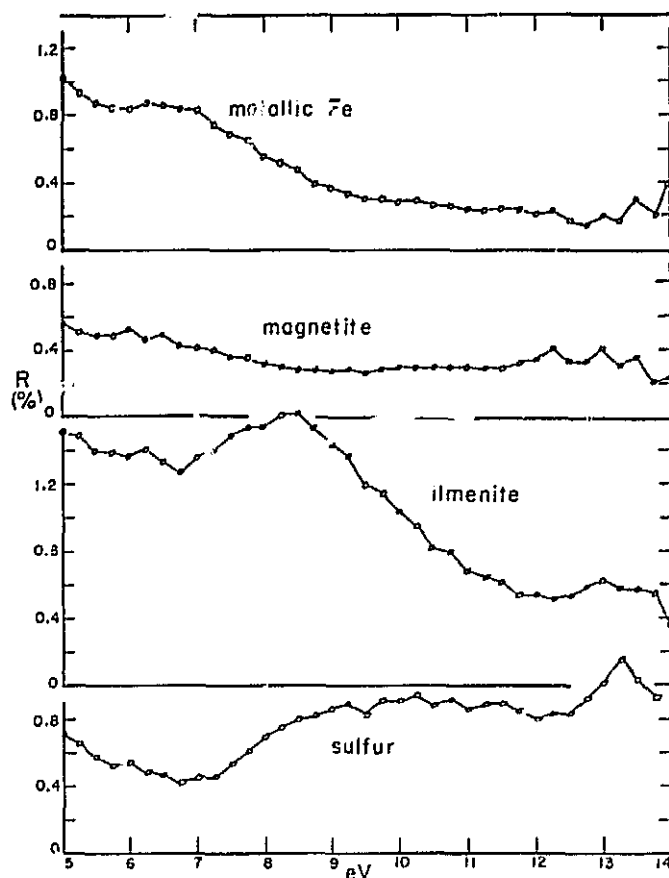


Fig. 6. Reflectance spectra vs. photon energy for three opaque minerals, plus sulfur (powders). The samples are: metallic Fe, synthetic carbonyl iron; synthetic magnetite; ilmenite, Ilmen Mts., USSR; reagent-grade sulfur.

The An peak of the Apollo 17 rock has a shoulder on its low energy side caused by ilmenite. The spectral features of Apollo 11 soil 10084 are more subdued than the rock spectra, showing that the poorly-understood lunar darkening process also affects the reflectance of the soil in the VUV. However, the augite maximum at 10.25 eV and the unseparated An/Il peak between 8 and 9 eV can still be recognized.

Figures 8 and 9 show the reflectance spectra of powdered lunar materials over the range of wavelengths between the far-UV through the near-IR. The spectra in these figures and in Table I are given in terms of wavelength, rather than eV, for ease of comparison between these results and the large body of work done at longer wavelengths. The spectra are plotted on a log-log display in order to show

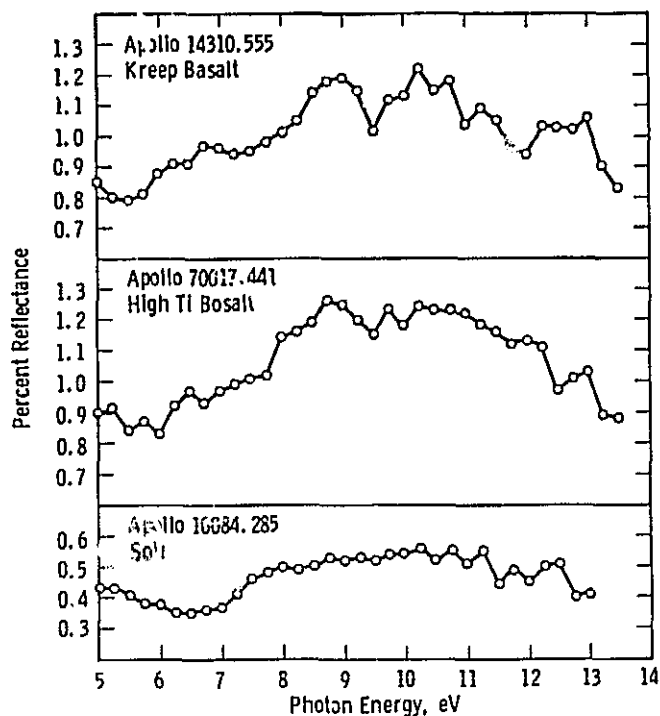


Fig. 7. Reflectance spectra vs. photon energy for three Apollo lunar samples: 14310, a KREEP basalt (rough chip); 70017, a high-Ti basalt (rough chip); 16084, a high-Ti lunar soil (powder).

Table 1. Absorption features in lunar rocks (0.09–1.80 μm)

Wavelength (μm)	Tentative Identification	Mineral	Reference
0.12	exciton	Augite	this work
0.14	exciton	Anorthite	this work
0.145		Ilmenite	this work
0.25	$\text{Fe}^{+2}\text{-O}^{+2}$ charge-transfer	Augite, Anorthite	(1)
0.34	$\text{Fe}^{+2}\text{-Ti}^{+4}$ charge-transfer	Augite	(1)
0.55		Ilmenite	(2), (3)
0.55	Ti^{+4} d-d	Augite	(4), (5)
0.95	Fe^{+2} d-d	Augite	(3), (4), (5)
1.25	Fe^{+2} d-d	Anorthite	(3), (5)
1.25		Ilmenite	(3)
1.70		Ilmenite	(3)

References:

- (1) Wells and Hapke (1977) (4) Burns (1970)
 (2) Loeffler *et al.* (1975) (5) Adams and McCord (1970)
 (3) Adams (1975)

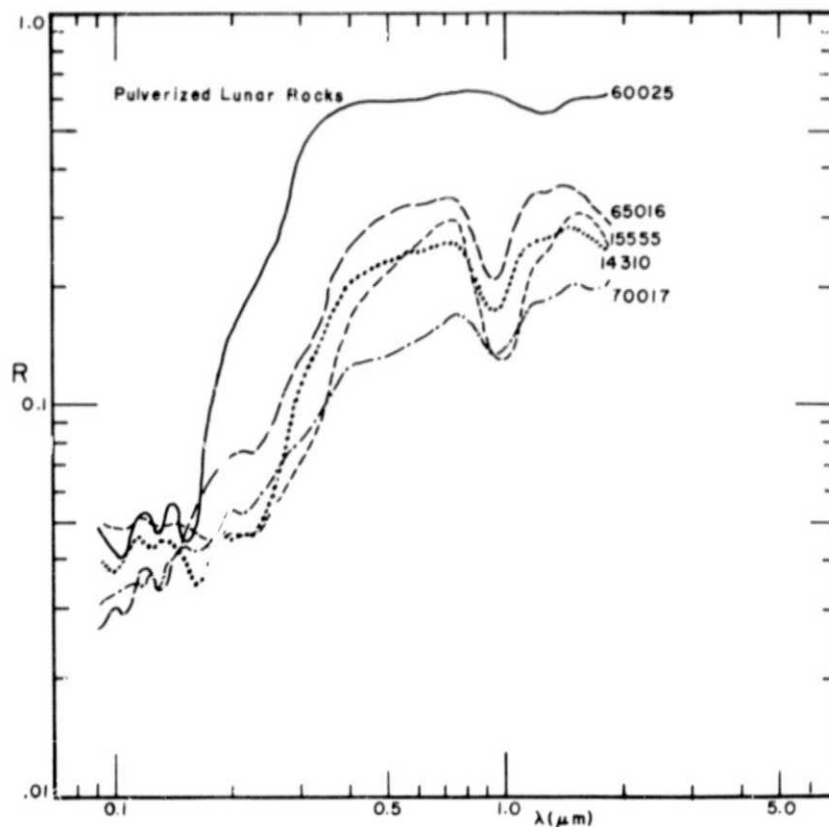


Fig. 8. Reflectance spectra vs. wavelength of several lunar crystalline rocks (pulverized to finer than $74\text{ }\mu\text{m}$). The samples are: 60025 (anorthosite), 65016 (anorthositic gabbro), 15555 (low-Ti basalt), 14310 (KREEP basalt) and 70017 (high-Ti basalt).

the various features over wide ranges in both wavelength and reflectance. The spectra were obtained by using a Cary 14 spectrophotometer to measure the bi-directional reflectance of loose powder over the range 0.20 to $1.80\text{ }\mu\text{m}$, and the vacuum spectrometer to measure pressed powder below $0.25\text{ }\mu\text{m}$. It was assumed that the relative VUV spectrum would not be appreciably affected by changes in packing, and the spectra were joined smoothly in the region of overlap. The spectra were normalized to their visual normal albedo relative to a BaSO_4 standard measured at a phase angle of 5° using a goniometric photometer with a narrow-band interference filter centered at $0.55\text{ }\mu\text{m}$.

Table I lists the various absorption bands which can be seen in the spectra in Fig. 8 and their identification. It is of interest to note that 65016 and 14310 both have similar major mineral content ($\sim 70\%$ Plag + 30% Pyr), but their spectra below about $0.31\text{ }\mu\text{m}$ are quite different. At present we are uncertain of the reason for

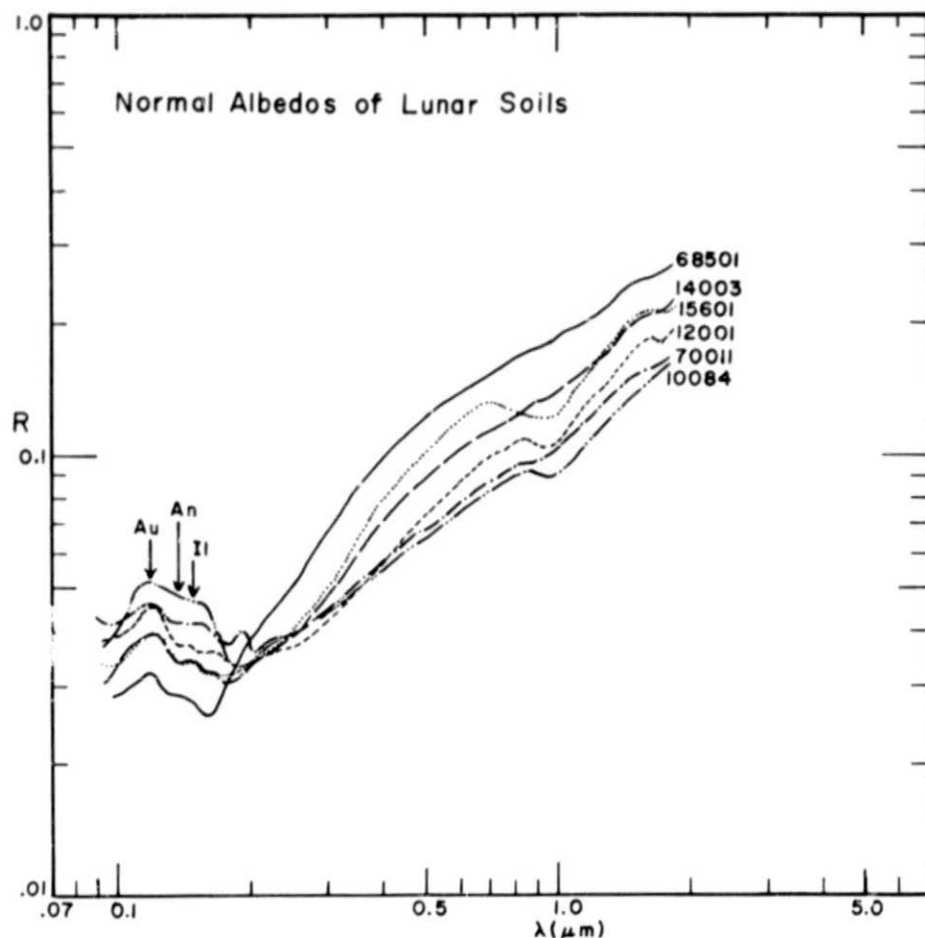


Fig. 9. Reflectance spectra vs. wavelength of six Apollo lunar fines.

these differences, but they do illustrate the ability of far-UV spectral measurements to distinguish between rock types.

Figure 9 shows the spectral reversal of lunar soils first observed on the moon by Lucke *et al.* (1974) using Apollo 17 data. Soils which are lighter in the visible and near IR are darker in the VUV. This inverse relationship characterizes only the soils and not the powdered lunar rocks. These spectra are consistent with earlier results of Lucke *et al.* (1973). The spectra of the soils have minima between 0.16 and 0.20 μm , with the most iron-poor soils having their minima at the shortest wavelengths. All the soils clearly show the augite peak at 0.12 μm and the An/Il shoulder at 0.15 μm . By contrast, few of the long-wavelength bands displayed by the rocks in Fig. 8 and Table I are visible in the soils. This again emphasizes the potential of the VUV for remote sensing.

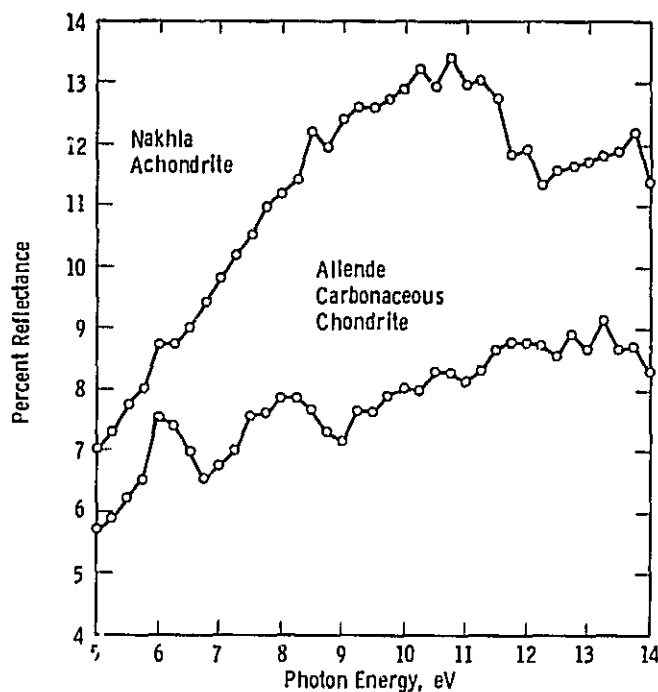


Fig. 10. Reflectance vs. photon energy for two meteorites (polished surfaces).

Figure 6 compares the spectra of three common opaque minerals, metallic iron, magnetite and ilmenite. Their spectra are quite different from each other, in contrast to their behavior at longer wavelengths where it is rather difficult to distinguish between them. Also shown in this figure is elemental sulfur, which is of interest in connection with the surfaces of outer planet satellites. Sulfur is unusual among the substances considered here in having its main peak at a quite high energy, 13.25 eV.

Finally, in Fig. 10 we show the spectra of two meteorites, Nakhla, an achondrite, and Allende, a type III carbonaceous chondrite. The achondrite clearly shows the 10.25 eV augite band, while Allende shows the 8.25 eV olivine band. Allende has a second band at 6.0 eV, which is unidentified, but which we speculate may be due to spinel.

CONCLUSIONS

The reflectance spectra of materials of geological interest have characteristic absorption peaks in the VUV region. We have shown that these peaks are visible when the surface is a powder as well as when it is polished or roughened. We are

presently measuring the spectra of salts and frosts, as well as meteorites and terrestrial minerals. It may be anticipated that the reflectances of solar system objects in the VUV will be an important new source of compositional information.

Acknowledgments—We thank Dr. W. J. Choyke for invaluable advice, help, encouragement and use of some of his equipment during the course of these measurements. B. Hapke thanks W. Fastie for interesting him in the problem of the VUV reflectance of the moon. This research is sponsored by a grant from the National Aeronautics and Space Administration, Lunar and Planetary Programs Office.

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VACUUM ULTRAVIOLET SPECTRA OF CARBONACEOUS CHONDRITES
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This paper on carbonaceous chondrites is the first in a survey of the vacuum ultraviolet reflection spectra of all types of meteorites. It is hoped that this investigation will be useful in the remote identification of asteroidal compositions. Investigation of the spectra of various exotic ices is also being undertaken. The region under study extends from 5 to 14 electron volts (ev) or 250-90 nm.

In the two major forms of silicates found in carbonaceous chondrites, olivines and pyroxenes, the unpolarized specular reflectance spectra can be divided into two major types, oxygen-transition metal ion charge-transfer at lower energy than the energy gap (E_g) excitation, and the latter. The energy gap excitation is related to the crystal structure, decreasing in energy inversely as the volume of the unit cell. Thus in olivine the E_g was found to vary from 7.4 ev in fayalite to 8.6 ev in forsterite (Nilsen and Shankland, 1976).

Figure 1 compares the reflectance of a polished (010) face of a gem peridot single crystal with that of a powdered crystal from the same locality, Sundalen, Norway. This olivine with E_g of 8.3 ev from the polished (010)

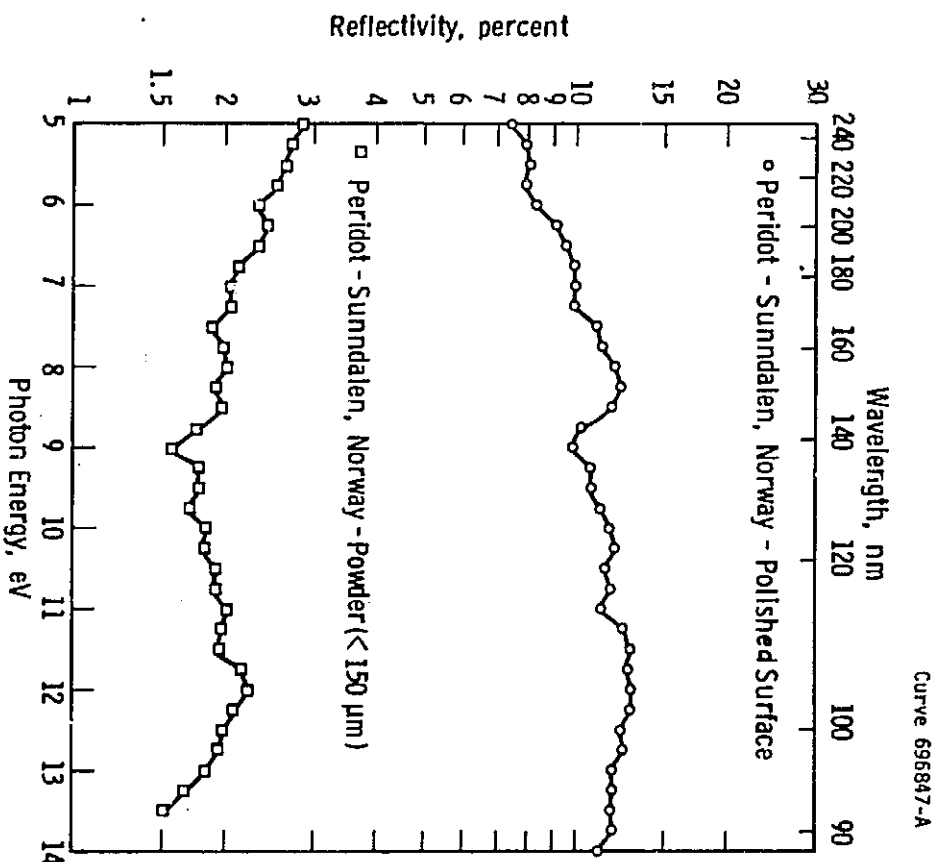


Fig. 1 Reflection spectra of Sundalen, Norway, forsterite peridot crystal on (010) face and powdered crystal.

surface and 8.25 eV from (001), is near the forsterite composition while Rockport, Mass. fayalite (Fig. 2) was found with an exceptionally low E_g of 7.0. Comparison of these two olivines indicates not only a shift in E_g but a

Curve 696859-A

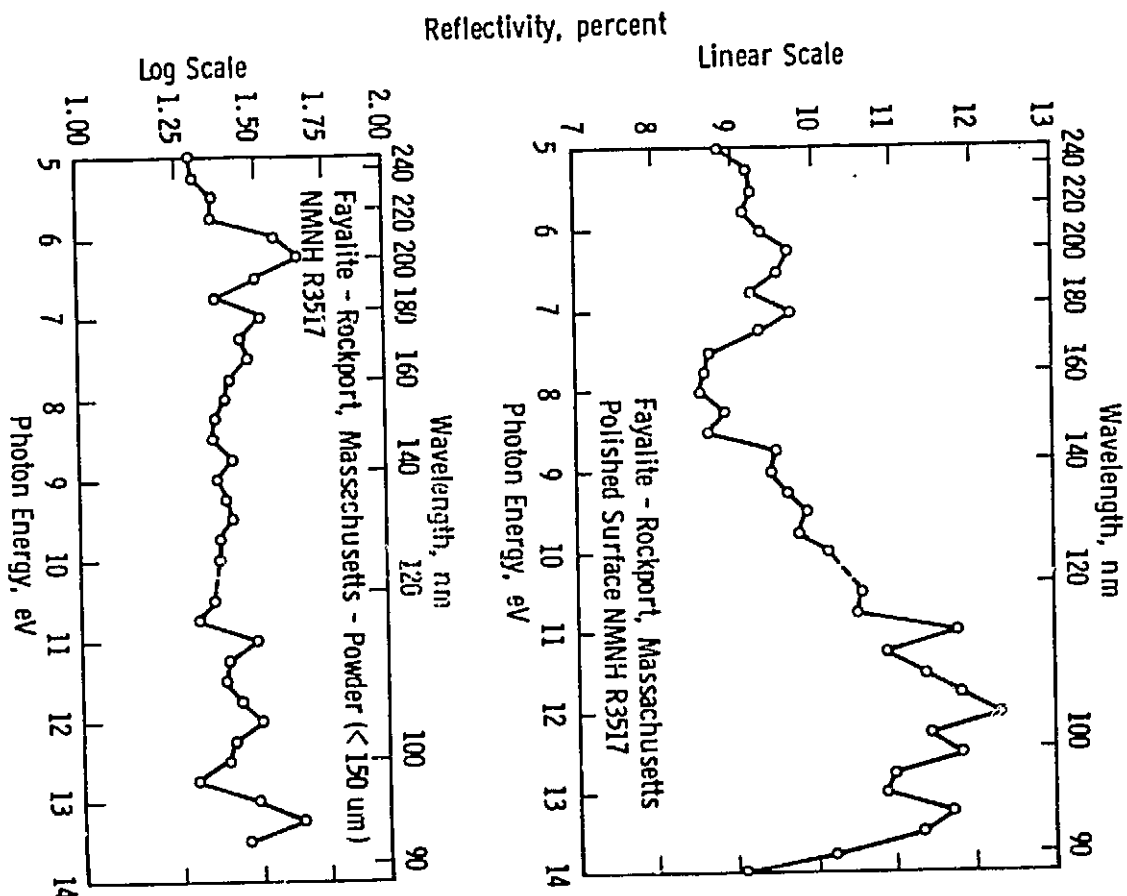


Fig. 2 Reflection spectra of polished Rockport, Mass. fayalite and powder.

corresponding shift in all the excitation bands of higher energy than E_g . The data for Orgueil (C-1) (Fig. 3) with E_g 's at 7.8 and 8.5 in the powder indicate variable olivine content as is the case.

Norton County, essentially iron-free enstatite, gave an E_g of 8.75 eV from a polished surface while the powder gave 8.5 eV. The powder

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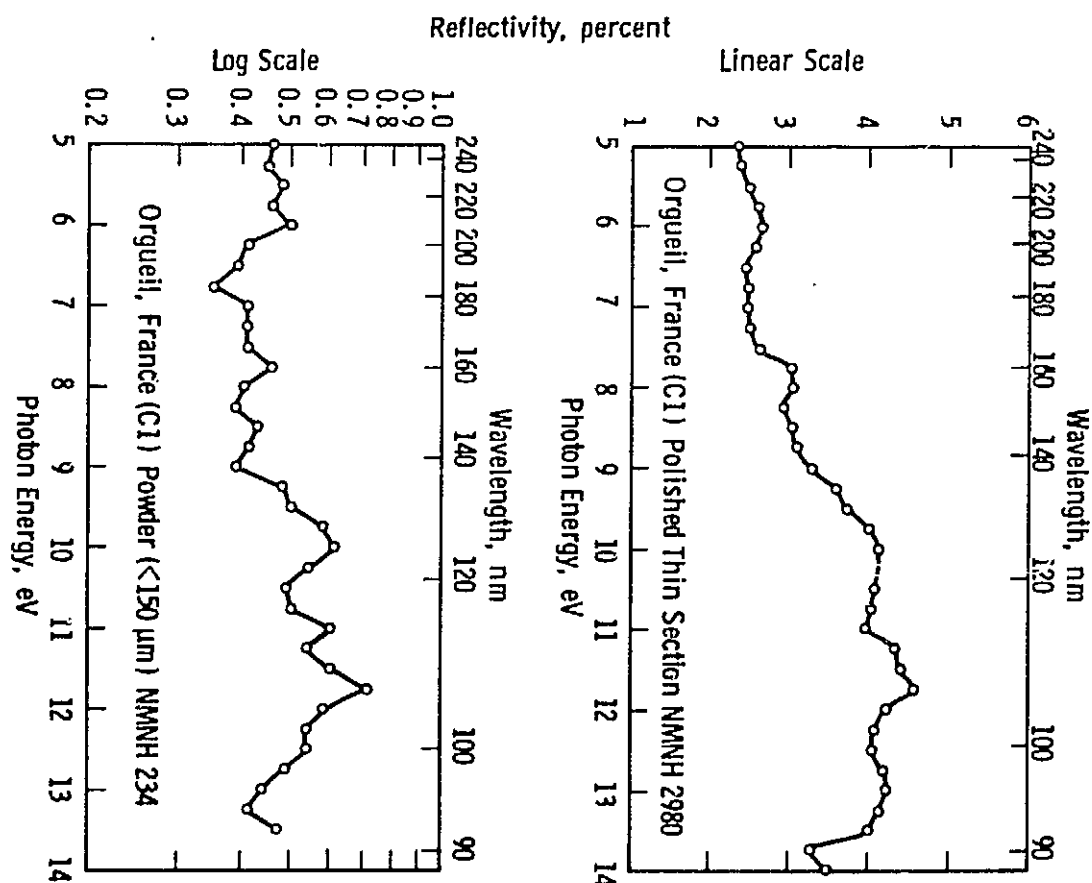


Fig. 3 Reflection spectra from surface of polished thin-section of Orgueil and from powder.

(< 150 nm) not only reflects better mixing of the constituent material but averaging of the anisotropic peak variation.

Tatohvne (FeO, 13.89%) a mononict breccia of hypersthene gave an Eg of 8.25 ev for both polished surface and powder. This value is consistent with the Norton County value. The polished thin-sections used had the minerals less randomized than in the powder on a micro-scale.

Angra dos Reis (Fe₂O₃, 2.89%, FeO, 4.1%) (Fig. 4) seen as a fassite polished standard gave an Eg value of 8.0 ev.

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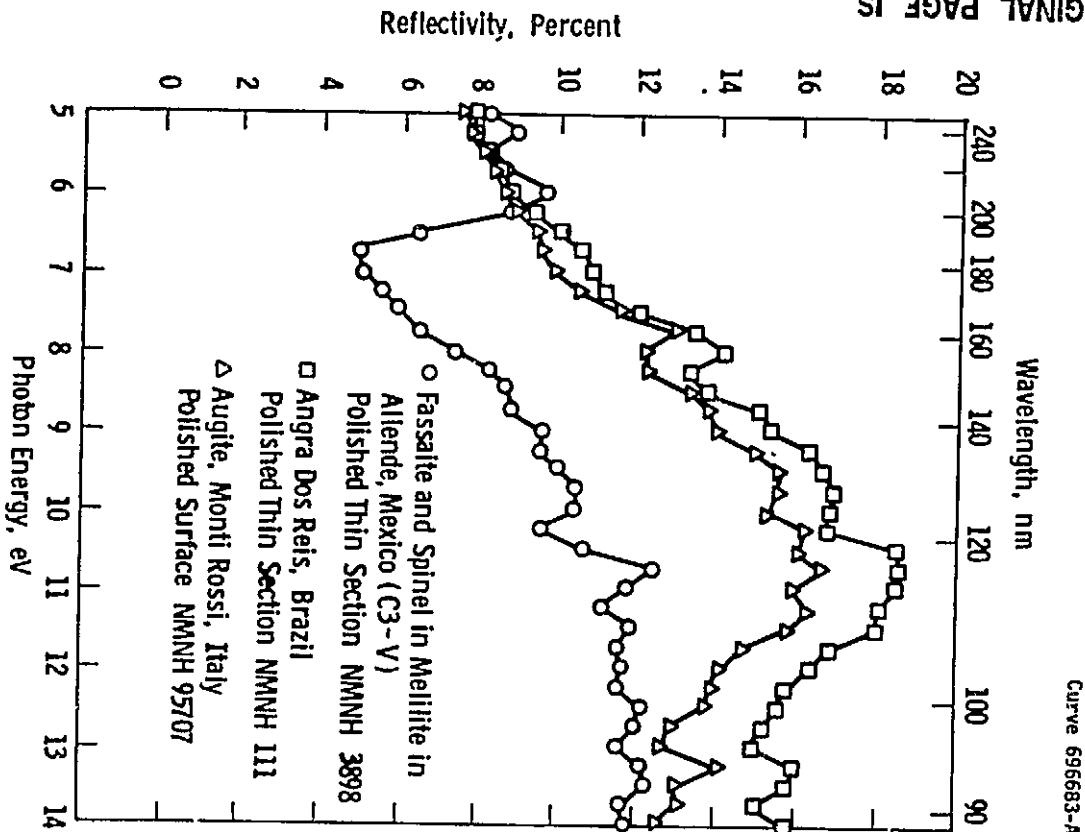


Fig. 4 Reflection spectra from meteoritic fassites and terrestrial augite.

Three augite standards were studied, Monte Rossi, Italy (Fig. 5) (Fe₂O₃, 4.17%, FeO, 5.98%), Le Rozier, France (Fe₂O₃, 3.60%, FeO, 4.90%), and Maui, Hawaii (Fe₂O₃, 3.36%, FeO, 4.43%) with the following Eg's respectively for polished surfaces 7.75 ev, 7.75 ev, and 8.0 ev. For the augite powders the values of Eg were 8.0 for both Monte Rossi and Maui. These Eg's

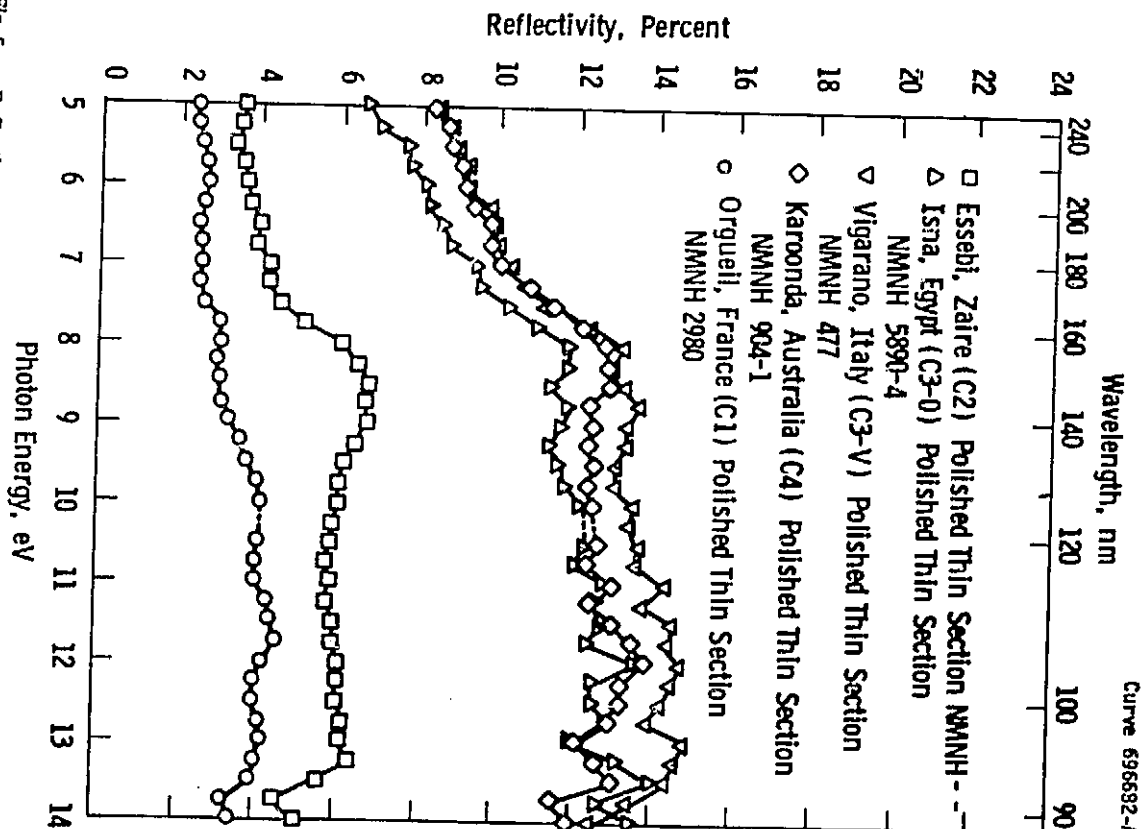


Fig. 5 Reflection spectra from polished thin-sections of a representative of each type of carbonaceous chondrite.

are in excellent agreement in regard to iron content. The spectra indicated the presence of hematite in the last two augites. Another intense excitation band gave values of 10.75 and 11.0 eV for the Monte Rossi and Maui powders. Thus excitation energy shifts in augites to lower energies related to increased iron content with larger cation size than magnesium are clearly indicated. This

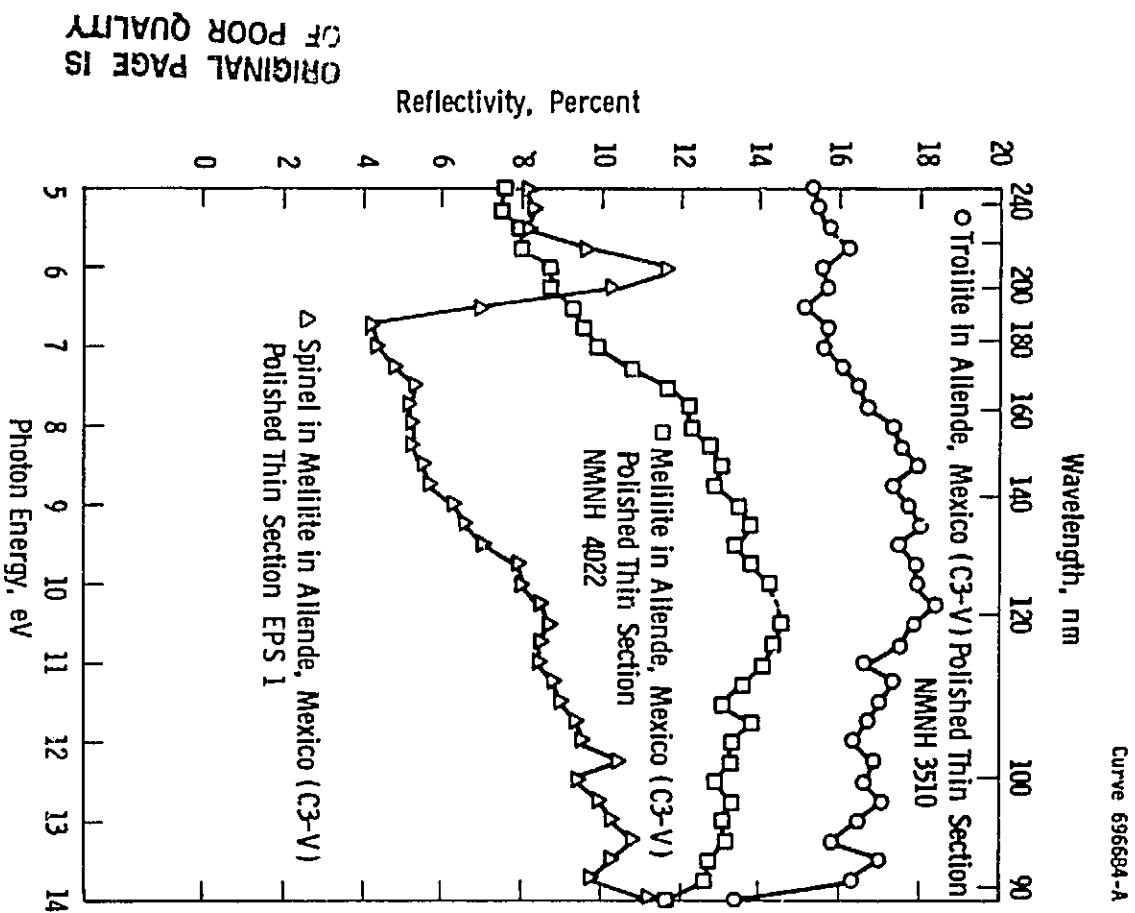


Fig. 6 Reflection spectra from various minerals in thin-sections of Allende.

intense excitation of the standard augites was at 10.75 eV in Angra dos Reis and Allende Fassite. The Eg of the latter was not resolved (Fig. 4).

The spectra of the different types of carbonaceous chondrites are illustrated in Figure 5. In addition to those illustrated in Figures 3, 4, and 5, polished thin-section reflection spectra were taken of the C-2 chondrites, Bells, Mighei, and Murchison; C-3(0), Kainsaz and Warrenton; and powder data was taken of Essebi, Murchison, Warrenton, and Allende.

Other polished and powdered analyzed standards were synthetic and natural specimens of spinel, ilmenite and hematite. A lunar anorthite was also used as a standard.

A melilita chondrule free of spinel gave an Eg at 8.5 eV as shown in Figure 6, NMNH 4022. An intense band at 6.0 eV related to the spinel in melilita in Allende, is shown in the same figure. This has been found in synthetic spinels containing iron and chromium. This band is also present in the spectra of Orgueil, Bells, and Murchison indicating the presence of Cr-spinel in these meteorites. Because of the unusually large intensity of this band, it is being investigated in detail.

The Eg's from polished surfaces of the C-2 chondrites, Bells, Essebi, Mighei, and Murchison were 8.75 eV, 8.50 eV, 8.25 eV, and 8.25 eV, respectively. They all exhibit intense excitation peaks at 11.0 eV and 13.25 eV and 13.25 eV which are characteristic of hematite. The C-3(0) polished surfaces of Isna, Kainsaz and Warrenton gave Eg's respectively of 8.25 eV, 8.0 eV and 8.0 eV. They exhibit a charge transfer peak between 7.00 and 7.13 eV that was found in no other materials except Bells, Essebi, Allende and Sundalen forsteritic olivine. The three C-3(0)'s also had a peak at 10.25 eV characteristic of the same olivine.

The C-3(V) chondrites Allende and Vigarano (Fig. 5) had Eg's at 8.00 eV and 8.13 eV. Vigarano did not show the spinel-related peak found on some of the surfaces of Allende studied. In order for polished surfaces and powders of Allende to both show the spinel-related peak, the powder sample must be taken next to the region where the thin-section sample containing spinel is removed.

Angra dos Reis and Allende fassite in polished section (Fig. 4) had an excitation band at 10.75 eV in common that also occurred in Warrenton and in the Le Rozier and Monti Rossi augite.

The C-4, Karoonda (Fig. 5), exhibited an Eg peak of 8.25 eV from the polished surface and 7.75 eV from the powder. This indicates an olivine content similar to the Sundalen peridot. The powder data could indicate a somewhat higher iron content in the Karoonda olivine.

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